

# **STIC Search Report**

## **Biotech-Chem Library**

**STIC Database Tracking Number: 175358**

**TO: Ben Sackey**  
**Location: 5b31 / 5c18**  
**Art Unit: 1626**  
**Friday, January 06, 2006**

**Case Serial Number: 10 / 705659**

**From: Noble Jarrell**  
**Location: Biotech-Chem Library**  
**Rem 1B71**  
**Phone: 272-2556**

**Noble.jarrell@uspto.gov**

### **Search Notes**

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175358

ACCESS DB #

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Scientific and Technical Information Center

## SEARCH REQUEST FORM

Requester's Full Name: BEN SACKETT Examiner #: 73489 Date: 12/20/05  
Art Unit: 1626 Phone Number: 2-0704 Serial Number: 101705659  
Location (Bldg/Room#): Ben 533 (Mailbox #): \_\_\_\_\_ Results Format Preferred (circle): PAPER DISK  
\*\*\*\*\*

To ensure an efficient and quality search, please attach a copy of the cover sheet, claims, and abstract or fill out the following:

Title of Invention: Compounds and Synthesis process.Inventors (please provide full names): William J. BegleyEarliest Priority Date: 11/10/03

## Search Topic:

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc., if known.

\*For Sequence Searches Only\* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

A process for preparing 6-chloro-2,5-dicarboxamide phenol comprising chlorinating 2-alkyl-6-amino-benzoxazole then to form 2-alkyl-6-amino-7-chlorobenzoxazole, reacting with an acid chloride and a base.

Thanks

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DEC 30 2005  
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## STAFF USE ONLY

Searcher: noble

Searcher Phone #: \_\_\_\_\_

Searcher Location: \_\_\_\_\_

Date Searcher Picked Up: 11/6/06Date Completed: 11/6/06Searcher Prep & Review Time: 10Online Time: 30

## Type of Search

\_\_\_\_ NA Sequence (#)

\_\_\_\_ AA Sequence (#)

5 Structure (#)✓ Bibliographic

\_\_\_\_ Litigation

\_\_\_\_ Fulltext

\_\_\_\_ Other

## Vendors and cost where applicable

✓ STN \_\_\_\_\_ Dialog

\_\_\_\_ Questel/Orbit \_\_\_\_\_ Lexis/Nexis

\_\_\_\_ Westlaw \_\_\_\_\_ WWW/Internet

\_\_\_\_ In-house sequence systems

\_\_\_\_ Commercial \_\_\_\_\_ Oligomer \_\_\_\_\_ Score/Length

\_\_\_\_ Interference \_\_\_\_\_ SPDI \_\_\_\_\_ Encode/Transl

\_\_\_\_ Other (specify)

=> b reg

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STRUCTURE FILE UPDATES: 4 JAN 2006 HIGHEST RN 871209-00-6  
 DICTIONARY FILE UPDATES: 4 JAN 2006 HIGHEST RN 871209-00-6

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TSCA INFORMATION NOW CURRENT THROUGH JULY 14, 2005

Please note that search-term pricing does apply when  
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\*\*\*\*\*  
 \*  
 \* The CA roles and document type information have been removed from \*  
 \* the IDE default display format and the ED field has been added, \*  
 \* effective March 20, 2005. A new display format, IDERL, is now \*  
 \* available and contains the CA role and document type information. \*  
 \*  
 \*\*\*\*\*

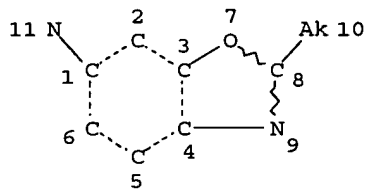
Structure search iteration limits have been increased. See HELP SLIMITS  
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REGISTRY includes numerically searchable data for experimental and  
 predicted properties as well as tags indicating availability of  
 experimental property data in the original document. For information  
 on property searching in REGISTRY, refer to:

<http://www.cas.org/ONLINE/UG/regprops.html>

=> d que sta l10

L5 STR



NODE ATTRIBUTES:  
 DEFAULT MLEVEL IS ATOM  
 DEFAULT ECLEVEL IS LIMITED

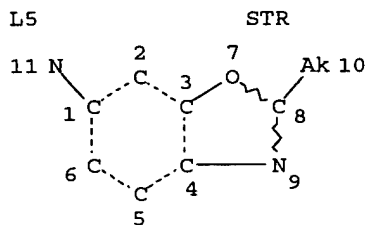
GRAPH ATTRIBUTES:  
 RING(S) ARE ISOLATED OR EMBEDDED  
 NUMBER OF NODES IS 11

STEREO ATTRIBUTES: NONE  
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100.0% PROCESSED 4059 ITERATIONS  
 SEARCH TIME: 00.00.01

632 ANSWERS

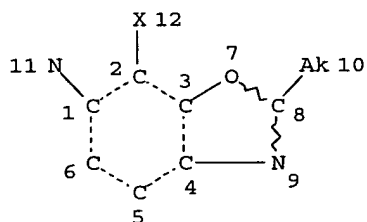
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NODE ATTRIBUTES:  
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GRAPH ATTRIBUTES:  
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 NUMBER OF NODES IS 11

STEREO ATTRIBUTES: NONE  
 L10 632 SEA FILE=REGISTRY SSS FUL L5  
 L11 STR



NODE ATTRIBUTES:  
 DEFAULT MLEVEL IS ATOM  
 DEFAULT ECLEVEL IS LIMITED

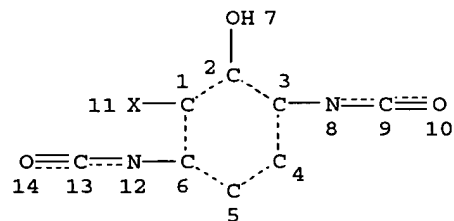
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 NUMBER OF NODES IS 12

STEREO ATTRIBUTES: NONE  
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100.0% PROCESSED 126 ITERATIONS  
 SEARCH TIME: 00.00.01

7 ANSWERS

=> d que sta l16  
 L14 STR



NODE ATTRIBUTES:  
 DEFAULT MLEVEL IS ATOM  
 DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED  
NUMBER OF NODES IS 14

STEREO ATTRIBUTES: NONE  
L16 68 SEA FILE=REGISTRY SSS FUL L14

100.0% PROCESSED 3134 ITERATIONS 68 ANSWERS  
SEARCH TIME: 00.00.01

=> b hcap  
FILE 'HCAPLUS' ENTERED AT 08:52:55 ON 06 JAN 2006  
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FILE COVERS 1907 - 6 Jan 2006 VOL 144 ISS 2  
FILE LAST UPDATED: 4 Jan 2006 (20060104/ED)

New CAS Information Use Policies, enter HELP USAGETERMS for details.

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> d all hitstr 124 tot

L24 ANSWER 1 OF 3 HCAPLUS COPYRIGHT 2006 ACS on STN  
AN 2005:411078 HCAPLUS  
DN 142:463458  
ED Entered STN: 13 May 2005  
TI Process for preparing 6-chloro-2,5-dicarbonamidophenol compounds  
IN Begley, William J.  
PA Eastman Kodak Company, USA  
SO U.S. Pat. Appl. Publ., 9 pp.  
CODEN: USXXCO  
DT Patent  
LA English  
IC ICM C07D-0263/52  
ICS C07C-0231/10  
INCL 548217000; 564155000  
CC 25-19 (Benzene, Its Derivatives, and Condensed Benzenoid Compounds)  
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US2005101784	A1	20050512	2003US-0705659	20031110
	WO2005047271	A1	20050526	2004WO-US36261	20041029
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW:	BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,				

EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,  
SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE,  
SN, TD, TG

PRAI 2003US-0705659 A 20031110

## CLASS

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
US 2005101784	ICM	C07D-0263/52
	ICS	C07C-0231/10
	INCL	548217000; 564155000
	IPCI	C07D0263-52 [ICM,7]; C07C0231-10 [ICS,7]
	NCL	548/217.000
	ECLA	C07C315/04; C07D263/56B
WO2005047271	IPCI	C07D0263-56 [ICM,7]; C07C0317-22 [ICS,7]
	ECLA	C07C315/04; C07D263/56B

AB Disclosed is a process for preparing a 6-chloro-2,5-dicarbonamidophenol compds. comprising a step employing a 2-alkyl-6-aminobenzoxazole to form a 2-alkyl-6-amino-7-chlorobenzoxazole in which the 2-alkyl group is unbranched at the  $\alpha$ -carbon. It also provides intermediate compds. useful in the process. The process provides a simple and safe way to prepare 6-chloro-2,5-dicarbonamidophenol compds. in good yield. Thus, nitration of 5-chloro-2-methylbenzoxazole by HNO<sub>3</sub>/H<sub>2</sub>SO<sub>4</sub> at 20° gave 5-chloro-2-methyl-6-nitrobenzoxazole which was reduced over Raney nickel in THF at room temperature under H pressure of 50 psi to give 6-Amino-5-chloro-2-methylbenzoxazole (I). Chlorination of I by sulfuryl chloride in EtOAc for 1 h gave 6-amino-5,7-dichloro-2-methylbenzoxazole which was acylated by 2-[(4-dodecyloxyphenyl)sulfonyl]butanoyl chloride in pyridine/EtOAc/THF at 15° for 30 min to give 6-[[2-[(4-dodecyloxyphenyl)sulfonyl]butanoyl]amino]-5,7-dichloro-2-methylbenzoxazole (II). Hydrolysis of II in a mixture of concentrated HCl and THF at 65° for .apprx.3 h gave 6-amino-2,4-dichloro-3-[[2-[(4-dodecyloxyphenyl)sulfonyl]butanoyl]amino]phenol which was acylated by 3,4-dichlorobenzoyl chloride in pyridine/THF at room temperature for 30 min to give 2,4-dichloro-3-[[2-[(4-dodecyloxyphenyl)sulfonyl]butanoyl]amino]-6-(3,4-benzoylamino)phenol.

ST alkylaminochlorobenzoxazole prepn intermediate chlorodicarbonamidophenol; chlorodicarbonamidophenol prepn

IT 701-16-6, 5-Fluoro-2-methylbenzoxazole 3024-72-4, 3,4-Dichlorobenzoyl chloride 3282-30-2, Pivaloyl chloride 19219-99-9, 5-Chloro-2-methylbenzoxazole 851486-98-1, 2-[(4-Dodecyloxyphenyl)sulfonyl]butanoic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(process for preparing 6-chloro-2,5-dicarbonamidophenol compds. by N-acylation of alkyl(amino)benzoxazole derivs. followed by hydrolysis and N-acylation)

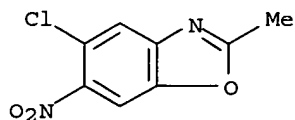
IT 13452-16-9P, 5-Chloro-2-methyl-6-nitrobenzoxazole  
40703-40-0P, 5-Fluoro-2-methyl-6-nitrobenzoxazole 98121-18-7P,  
2-[(4-Dodecyloxyphenyl)sulfonyl]butanoyl chloride 323579-00-6P,  
6-Amino-5-chloro-2-methylbenzoxazole 851486-89-0P,  
6-Amino-5,7-dichloro-2-methylbenzoxazole 851486-90-3P,  
6-[[2-[(4-Dodecyloxyphenyl)sulfonyl]butanoyl]amino]-5,7-dichloro-2-methylbenzoxazole 851486-91-4P, 2,4-Dichloro-3-[[2-[(4-dodecyloxyphenyl)sulfonyl]butanoyl]amino]-6-aminophenol  
851486-92-5P, 2,4-Dichloro-3-[[2-[(4-dodecyloxyphenyl)sulfonyl]butanoyl]amino]-6-(3,4-dichlorobenzoylamino)phenol 851486-93-6P,  
6-Amino-5-fluoro-2-methylbenzoxazole 851486-94-7P,  
6-Amino-7-chloro-5-fluoro-2-methylbenzoxazole 851486-95-8P,  
6-[[2-[(4-Dodecyloxyphenyl)sulfonyl]butanoyl]amino]-7-chloro-5-fluoro-2-methylbenzoxazole 851486-96-9P, 6-Amino-2-chloro-3-[[2-[(4-dodecyloxyphenyl)sulfonyl]butanoyl]amino]-4-fluorophenol  
851486-97-0P, 2-Chloro-3-[[2-[(4-dodecyloxyphenyl)sulfonyl]butanoyl]amino]-4-fluoro-6-(pivaloylamino)phenol

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

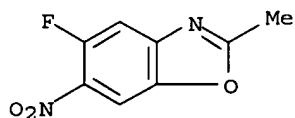
(process for preparing 6-chloro-2,5-dicarbonamidophenol compds. by N-acylation of alkyl(amino)benzoxazole derivs. followed by hydrolysis

and N-acylation)

IT 13452-16-9P, 5-Chloro-2-methyl-6-nitrobenzoxazole  
 40703-40-0P, 5-Fluoro-2-methyl-6-nitrobenzoxazole  
 323579-00-6P, 6-Amino-5-chloro-2-methylbenzoxazole  
 851486-89-0P, 6-Amino-5,7-dichloro-2-methylbenzoxazole  
 851486-90-3P, 6-[[2-[(4-Dodecyloxyphenyl)sulfonyl]butanoyl]amino]-  
 5,7-dichloro-2-methylbenzoxazole 851486-92-5P,  
 2,4-Dichloro-3-[[2-[(4-dodecyloxyphenyl)sulfonyl]butanoyl]amino]-6-(3,4-  
 dichlorobenzoylamino)phenol 851486-93-6P, 6-Amino-5-fluoro-2-  
 methylbenzoxazole 851486-94-7P, 6-Amino-7-chloro-5-fluoro-2-  
 methylbenzoxazole 851486-95-8P, 6-[[2-[(4-  
 Dodecyloxyphenyl)sulfonyl]butanoyl]amino]-7-chloro-5-fluoro-2-  
 methylbenzoxazole 851486-97-0P, 2-Chloro-3-[[2-[(4-  
 dodecyloxyphenyl)sulfonyl]butanoyl]amino]-4-fluoro-6-(pivaloylamino)phenol  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
 (Reactant or reagent)  
 (process for preparing 6-chloro-2,5-dicarbonamidophenol compds. by  
 N-acylation of alkyl(amino)benzoxazole derivs. followed by hydrolysis  
 and N-acylation)  
 RN 13452-16-9 HCAPLUS  
 CN Benzoxazole, 5-chloro-2-methyl-6-nitro- (8CI, 9CI) (CA INDEX NAME)

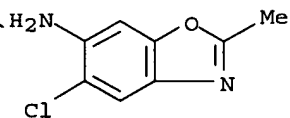


RN 40703-40-0 HCAPLUS  
 CN Benzoxazole, 5-fluoro-2-methyl-6-nitro- (9CI) (CA INDEX NAME)



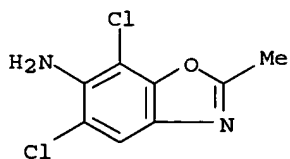
RN 323579-00-6 HCAPLUS  
 CN 6-Benzoxazolamine, 5-chloro-2-methyl- (9CI) (CA INDEX NAME)

nitro-methyl-  
 6-yl.



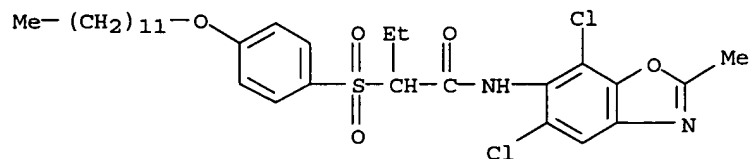
RN 851486-89-0 HCAPLUS  
 CN 6-Benzoxazolamine, 5,7-dichloro-2-methyl- (9CI) (CA INDEX NAME)

nitro-methyl-  
 6-yl.



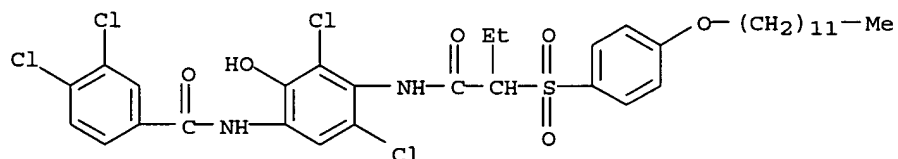
RN 851486-90-3 HCAPLUS  
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(dodecyloxy)phenyl]sulfonyl]- (9CI) (CA INDEX NAME)



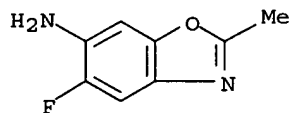
RN 851486-92-5 HCAPLUS

CN Benzamide, 3,4-dichloro-N-[3,5-dichloro-4-[[2-[[4-(dodecyloxy)phenyl]sulfonyl]-1-oxobutyl]amino]-2-hydroxyphenyl]- (9CI)  
(CA INDEX NAME)



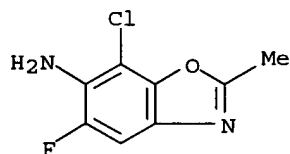
RN 851486-93-6 HCAPLUS

CN 6-Benzoxazolamine, 5-fluoro-2-methyl- (9CI) (CA INDEX NAME)



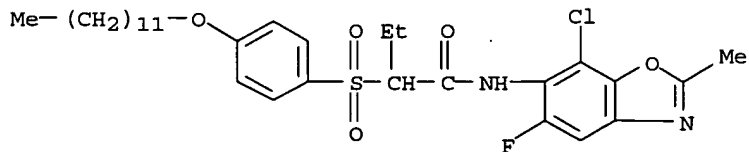
RN 851486-94-7 HCAPLUS

CN 6-Benzoxazolamine, 7-chloro-5-fluoro-2-methyl- (9CI) (CA INDEX NAME)



RN 851486-95-8 HCAPLUS

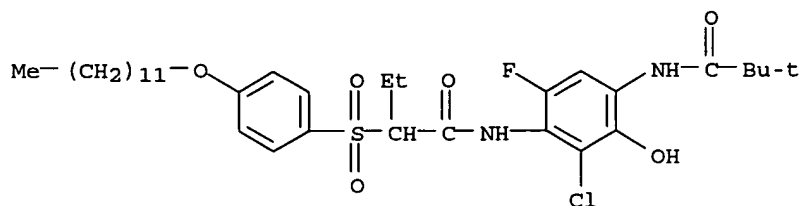
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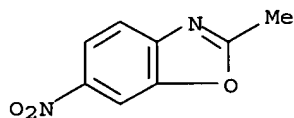
RN 851486-97-0 HCAPLUS

CN Butanamide, N-[2-chloro-4-[(2,2-dimethyl-1-oxopropyl)amino]-6-fluoro-3-hydroxyphenyl]-2-[[4-(dodecyloxy)phenyl]sulfonyl]- (9CI) (CA INDEX NAME)

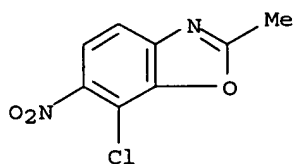




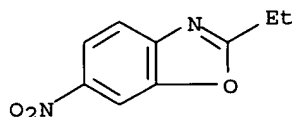
L24 ANSWER 2 OF 3 HCAPLUS COPYRIGHT 2006 ACS on STN  
 AN 1967:2504 HCAPLUS  
 DN 66:2504  
 ED Entered STN: 12 May 1984  
 TI Syntheses of heterocyclic compounds. XIV. Oxazoles from the pyrolysis of  
 AU aryl azides in a mixture of a carboxylic and polyphosphoric acid  
 CS Garner, Robert; Mullock, E. B.; Suschitzky, Hans  
 SO Roy. Coll. Advan. Technol., Salford, UK  
 SO Journal of the Chemical Society [Section] C: Organic (1966), (21), 1980-3  
 CODEN: JSOAX; ISSN: 0022-4952  
 DT Journal  
 LA English  
 CC 28 (Heterocyclic Compounds (More Than One Hetero Atom))  
 GI For diagram(s), see printed CA Issue.  
 AB cf. CA 65, 15366b. Aromatic azides (I) with a para-substituent decompose  
 thermally in a mixture of polyphosphoric and a carboxylic acid to give  
 oxazoles (II), or in some cases N,O-diacyl o-aminophenols, in good yield.  
 Various aspects of this nitrene mechanism are discussed. 18 references.  
 ST OXAZOLES BENZO; AZIDES; BENZOXAZOLES  
 IT Aryl azides  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (pyrolysis of)  
 IT 288-42-6D, Oxazole, derivs.  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (from azide pyrolysis)  
 IT 833-62-5P 5683-43-2P 13243-31-7P 13243-32-8P 13243-36-2P  
 13243-37-3P 13243-38-4P 13243-39-5P 13243-40-8P  
 13438-55-6P 13452-13-6P 13452-14-7P 13452-15-8P 13452-16-9P  
 13452-17-0P 13473-67-1P 14724-89-1P 15260-89-6P  
 34594-87-1P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 IT 5683-43-2P 13243-38-4P 13243-39-5P  
 13452-16-9P 13452-17-0P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 RN 5683-43-2 HCAPLUS  
 CN Benzoxazole, 2-methyl-6-nitro- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



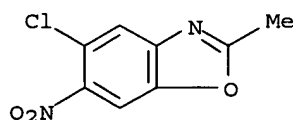
RN 13243-38-4 HCAPLUS  
 CN Benzoxazole, 7-chloro-2-methyl-6-nitro- (8CI) (CA INDEX NAME)



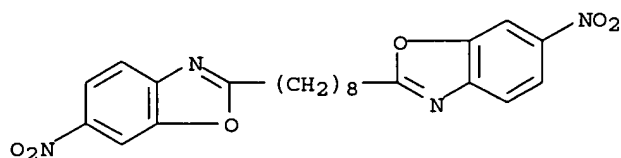
RN 13243-39-5 HCAPLUS  
CN Benzoxazole, 2-ethyl-6-nitro- (8CI) (CA INDEX NAME)



RN 13452-16-9 HCAPLUS  
CN Benzoxazole, 5-chloro-2-methyl-6-nitro- (8CI, 9CI) (CA INDEX NAME)



RN 13452-17-0 HCAPLUS  
CN Benzoxazole, 2,2'-octamethylenebis[6-nitro- (8CI) (CA INDEX NAME)



L24 ANSWER 3 OF 3 HCAPLUS COPYRIGHT 2006 ACS on STN  
AN 1953:41191 HCAPLUS  
DN 47:41191  
OREF 47:6894f-i,6895a-e  
ED Entered STN: 22 Apr 2001  
TI Quinone imides. XVIII. p-Quinonedipivalimides and their reactions  
AU Adams, Roger; Stewart, John M.  
CS Univ. of Illinois, Urbana  
SO Journal of the American Chemical Society (1952), 74, 3660-4  
CODEN: JACSAT; ISSN: 0002-7863  
DT Journal  
LA Unavailable  
CC 10 (Organic Chemistry)  
AB To 10.8 g. p-C6H4(NH2)2 (I) in 125 cc. pyridine was added slowly with stirring 25.3 g. Me3CCOCl (II) (obtained nearly quantitatively by refluxing the acid 1 hr. with excess SOCl2), and the mixture poured after 6 hrs. into excess HCl and ice to give 25.5 g. (93%) p-C6H4(NHCOCMe3)2, m. 283° (from dioxane). Similarly was prepared from 4.6 g. Ph3CCOCl (obtained by carbonation of Ph3CMgCl and treatment of the acid with SOCl2) in 20 cc. pyridine and 0.81 g. I in 10 cc. pyridine 4.5 g. (92.5%) p-C6H4(NHCOCPh3)2, m. 324-6° (from HCONMe2). I (5.0 g.) and 20 cc. CF3CO2H refluxed 7 hrs., and the mixture poured into 400 cc. dilute HCl

yielded 5.9 g. (38%) p-C<sub>6</sub>H<sub>4</sub>(NHCOCF<sub>3</sub>)<sub>2</sub>, m. 274° (from dioxane), which, refluxed with Pb(OAc)<sub>4</sub> in CHCl<sub>3</sub>, tarry, amorphous products. A similar oxidation of p-C<sub>6</sub>H<sub>4</sub>(NH-COCCl<sub>3</sub>)<sub>2</sub> gave a small yield of an unstable product. Equimolar amts. of N,N'-(p-phenylene)dipivalamides (III) and Pb(OAc)<sub>4</sub> refluxed 2 hrs. in dry CCl<sub>4</sub> (25 cc./g. III) yielded the corresponding substituted N,N'-dipivalyl-p-quinone diimines [substituent, m.p., % yield given]: H (V), 164.5°, 84; 2-Cl (VI), 81-8.5°, 65; 2,6-Cl<sub>2</sub> (VII), 104.5-6.5°, 85; 2,5-Cl<sub>2</sub> (VIII), 159.5-60.5°, 80; 2,3-Cl<sub>2</sub> (IX), 138.5-9.5°, 75; 2,3,5-Cl<sub>3</sub> (X), 115.5°, 78 (all recrystd. from petr. ether), p-C<sub>6</sub>H<sub>4</sub>(NHCOCMe<sub>3</sub>)<sub>2</sub> (0.83 g.) and an equivalent amount of Pb(OAc)<sub>4</sub> in Ac<sub>2</sub>O stirred 24 hrs. at 70° and the mix decomposed with 500 cc. H<sub>2</sub>O gave 31% 2,3,5,6-tetrachloro-IV, m. 200.5-1°. HCl passed into a petr. ether solution of the IV precipitated Cl-substituted III (substituent, m.p., and yield given): 2-Cl, 215°, 90; 2,6-Cl<sub>2</sub> (XI), 257°, 95, from VI; 2,3,5-Cl<sub>3</sub> (XII), m. 205-6° with rapid shrinking at 196°, 96% from VIII, 97% from IX, and 24% from VII (all from CHCl<sub>3</sub>-petr. ether); and 2,3,5,6-Cl<sub>4</sub> (XIII), m. 335-5.5° (from CHCl<sub>3</sub>), 44 yield together with 45% 2-tert-butyl-4,5,7-trichloro-6-(pivalylamino)benzoxazole, m. 225° (from aqueous EtOH), from X. V (0.69 g.) in 10 cc. glacial AcOH let stand 1 day at room temperature and poured into H<sub>2</sub>O yielded 0.3 g. (36%) 2,1,4-AcOC<sub>6</sub>H<sub>3</sub>(NHCOCMe<sub>3</sub>)<sub>2</sub> (XIV), needles, m. 156.5-7° (from CHCl<sub>3</sub>). To 9.0 g. V added slowly with stirring and cooling 9 cc. 98% HCO<sub>2</sub>H, and the mixture diluted with cold Et<sub>2</sub>O yielded 8.5 g. (81%) 2,1,4-HCOC<sub>6</sub>H<sub>3</sub>(NHCOCMe<sub>3</sub>)<sub>2</sub> (XV), platelets, m. 200-1° (from CHCl<sub>3</sub>). XIV refluxed 30 min. with 10% aqueous NaOH gave 2,5-(Me<sub>3</sub>CCONH)2C<sub>6</sub>H<sub>3</sub>OH (XVI), needles, m. 248° (from petr. ether), also obtained in 90% yield by boiling 4.0 g. XV in 100 cc. (CH<sub>2</sub>OH)<sub>2</sub> 5 min., or in 77% yield by alkaline hydrolysis of XV. XVI (0.30 g.) heated 15 min. at 250° and 100 mm. pressure and the cooled melt triturated with 15 cc. Et<sub>2</sub>O yielded 0.18 g. (84%) 2-tert-butyl-6-(pivaloylamino)benzoxazole, platelets or needles, m. 164-5° (from Et<sub>2</sub>O-petr. ether), hydrolyzed to XVI by refluxing 3 hrs. with 10% aqueous NaOH. The following Cl-substituted III (substituent given) were prepared from the corresponding Cl-substituted 1.2HCl salts and II in pyridine: 2,5-Cl<sub>2</sub>, needles, m. 239-40° (from CHCl<sub>3</sub>-petr. ether); 2,3-Cl<sub>2</sub>, needles, 61%, m. 200-1° (from MeOH) [the free diamine, needles, m. 120.5-1° (from H<sub>2</sub>O)]; XI, m. 256-7°, in poor yield. HCl passed 10 min. into 7.0 g. VII in 250 cc. petr. ether and the product chromatographed from 8 l. 3:1 petr. ether-Et<sub>2</sub>O mixture on activated Al<sub>2</sub>O<sub>3</sub> gave 24% XII and 33% 2-tert-butyl-5,7-dichloro-6-(pivalylamino)benzoxazole (XVII), needles, m. 165.5-7.5° (from petr. ether). Aqueous alkaline hydrolysis of XVII yielded 88% 2,4,3,6-Cl<sub>2</sub>(Me<sub>3</sub>CCONH)2C<sub>6</sub>HOH (XVIII), needles, m. 226° (from Et<sub>2</sub>O-petr. ether). XVIII in (CH<sub>2</sub>OH)<sub>2</sub> boiled 10 min. gave XVII. Cl passed into 2.0 g. XVI in 100 cc. glacial AcOH at 20° to a weight increase of 0.95 g., and the mixture E poured into 600 cc. cold H<sub>2</sub>O gave XVIII. To 0.12 g. XVIII in 20 cc. H<sub>2</sub>O and 0.5 cc. 5% aqueous NaOH was added with stirring 0.04 cc. Ac<sub>2</sub>O to give 0.10 g. (83%) acetate of XVIII, m. 267-8° (from CHCl<sub>3</sub>-petr. ether). IX (0.3 g.) in 5 cc. 98% HCO<sub>2</sub>H let stand 0.5 hr. at room temperature, the red solution diluted with 75 cc. Et<sub>2</sub>O, extracted with 100 cc. 5% aqueous NaOH, and the extract acidified with HCl yielded 0.2 g. (63%) 3,4,2,5-Cl<sub>2</sub>(Me<sub>3</sub>CCONH)2C<sub>6</sub>HOH, m. 190.5-1° (from CHCl<sub>3</sub>-petr. ether). Similarly was prepared 3,6,2,5-Cl<sub>2</sub>(Me<sub>3</sub>CCONH)2C<sub>6</sub>HOH, 44%, m. 199.5-200.5° (from CHCl<sub>3</sub>-petr. ether), from VIII. p-C<sub>6</sub>Cl<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> (2.5 g.) and 2.5 g. II in 25 cc. pyridine refluxed 4.5 hrs. gave 4.0 g. (95%) XIII.

IT Quinone imines

IT Oxidation

(of N,N'-p-phenylenebis amides)

IT Propionamide, N,N'-[2,3-dichloro-3-5-hydroxy-p-phenylene]bis[2,2-dimethyl-

Propionamide, N,N'-[2,3-dichloro-5-5-hydroxy-p-phenylene]bis[2,2-dimethyl-

Propionamide, N,N'-[2,5-dichloro-3-5-hydroxy-p-phenylene]bis[2,2-dimethyl-

Propionamide, N,N'-[2,5-dichloro-5-5-hydroxy-p-phenylene]bis[2,2-dimethyl-

IT 859057-55-9, Propionamide, N,N'-(hydroxy-p-phenylene)bis[2,2-dimethyl-

(and esters)

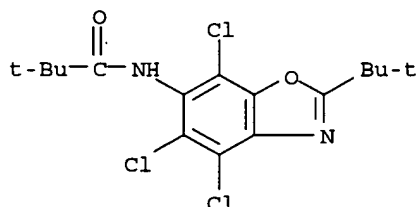
IT 4257-74-3, Acetamide, N,N'-p-phenylenebis[2,2,2-trichloro-

(oxidation of)

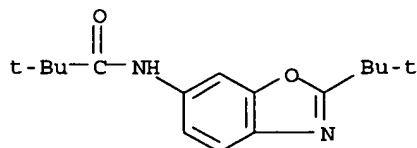
IT 404-28-4, Acetamide, N,N'-p-phenylenebis[2,2,2-trifluoro- 6068-70-8, Acetyl chloride, triphenyl- 6937-98-0, Propionamide, N,N'-p-phenylenebis[2,2-dimethyl- 41946-53-6, p-Phenylenediamine, 2,3-dichloro- 313394-34-2, Propionamide, N,N'-(chloro-p-phenylene)bis[2,2-dimethyl- 854052-01-0, p-Benzoquinone diimine, 2,3,5-trichloro-N,N'-dipivaloyl- 854052-02-1, p-Benzoquinone diimine, 2,3,5,6-tetrachloro-N,N'-dipivaloyl- 854052-25-8, p-Benzoquinone diimine, 2-chloro-N,N'-dipivaloyl- 854053-46-6, p-Benzoquinone diimine, 2,6-dichloro-N,N'-dipivaloyl- 854053-47-7, p-Benzoquinone diimine, 2,5-dichloro-N,N'-dipivaloyl- 854053-48-8, p-Benzoquinone diimine, 2,3-dichloro-N,N'-dipivaloyl- 854164-19-5, Benzoxazole, 2-tert-butyl-4,5,7-trichloro-6-pivalamido- 854164-19-5, Propionamide, N-(2-tert-butyl-4,5,7-trichloro-6-benzoxazolyl)-2,2-dimethyl- 854164-20-8, Benzoxazole, 2-tert-butyl-6-pivalamido- 854164-20-8, Propionamide, N-2-tert-butyl-6-benzoxazolyl-2,2-dimethyl- 854164-22-0, Benzoxazole, 2-tert-butyl-5,7-dichloro-6-pivalamido- 854164-22-0, Propionamide, N-(2-tert-butyl-5,7-dichloro-6-benzoxazolyl)-2,2-dimethyl- 855464-56-1, p-Benzoquinone diimine, N,N'-dipivaloyl- 856985-71-2, Propionamide, N,N'-(2,6-dichloro-p-phenylene)bis[2,2-dimethyl- 856985-73-4, Propionamide, N,N'-(2,5-dichloro-p-phenylene)bis[2,2-dimethyl- 856985-75-6, Propionamide, N,N'-(2,3-dichloro-p-phenylene)bis[2,2-dimethyl- 856985-79-0, Propionamide, N,N'-(3,5-dichloro-2-hydroxy-p-phenylene)bis[2,2-dimethyl-, acetate 857231-91-5, Propionamide, N,N'-(trichloro-p-phenylene)bis[2,2-dimethyl- 857943-06-7, Propionamide, N,N'-(tetrachloro-p-phenylene)bis[2,2-dimethyl- 859301-32-9, Propionamide, N,N'-(3,5-dichloro-2-hydroxy-p-phenylene)bis[2,2-dimethyl- 861058-90-4, Acetamide, N,N'-p-phenylenebis[2,2,2-triphenyl- (preparation of)

IT 854164-19-5, Benzoxazole, 2-tert-butyl-4,5,7-trichloro-6-pivalamido- 854164-20-8, Benzoxazole, 2-tert-butyl-6-pivalamido- 854164-22-0, Benzoxazole, 2-tert-butyl-5,7-dichloro-6-pivalamido- 859301-32-9, Propionamide, N,N'-(3,5-dichloro-2-hydroxy-p-phenylene)bis[2,2-dimethyl- (preparation of)

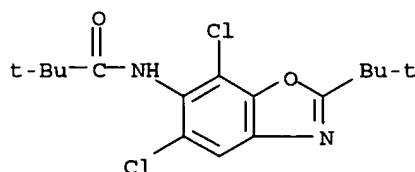
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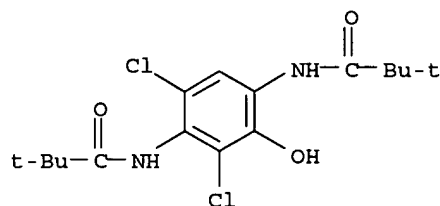
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RN 854164-22-0 HCAPLUS  
CN INDEX NAME NOT YET ASSIGNED



RN 859301-32-9 HCAPLUS  
 CN Propionamide, N,N'-(3,5-dichloro-2-hydroxy-p-phenylene)bis[2,2-dimethyl-  
 (5CI) (CA INDEX NAME)



*End product.*

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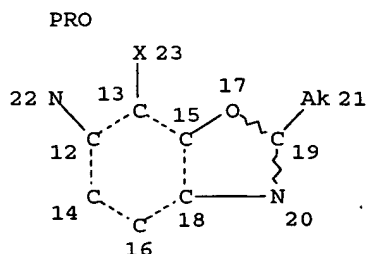
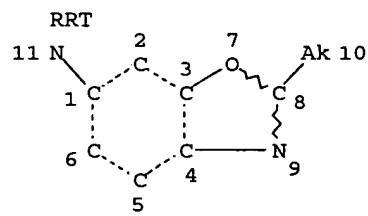
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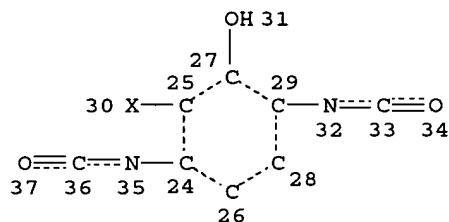
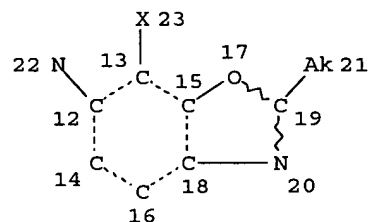
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RRT

PRO



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